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(54) ENGINE LUBRICATING OIL AND LUBRICATION

(57)Abstract:

PROBLEM TO BE SOLVED: To obtain a lubricating oil which remarkably reduces the friction resistance inside an engine thereby to reduce the noise caused by engine rotation, to improve fuel consumption, and to extend the life of the engine and which itself has a long life by dispersing an effective amt. of a fine BN powder having a crystalline disordered layer structure in an oil.

SOLUTION: An effective amt. of a fine BN powder having a crystalline disordered layer structure and pref. having an average primary particle size of 0.5 μm or lower is dispersed in an oil, pref. a petroleum-derived oil, or a synthetic oil, or a mixture of these oils. Alternatively, a fine BN powder of a crystalline disordered layer structure and a fine BN powder of a hexagonal system, both having an average primary particle size of 1 μm or lower, pref. 0.5 μm or lower, are dispersed in the oil, the amt. of the powder of a crystalline disordered layer structure accounting for at least 50 wt.% of the whole powder dispersed. The amt. of the whole powder dispersed is pref. 0.02-50 wt.%.

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* NOTICES *

1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

CLAIMS

[Claim(s)]
[Claim 1] Engine lubricant characterized by carrying out effective dose distribution and containing the boron nitride impalpable powder of crystalline random layer structure in an oil.
[Claim 2] Engine lubricant which the boron nitride impalpable powder of crystalline random layer structure 1 micrometer or less and the mean particle diameter of a primary particle distribute boron nitride impalpable powder with a hexagonal system of 1 micrometer or less, and the mean particle diameter of a primary particle contains in an oil, and is characterized by 50% of the weight or more of boron nitride impalpable powder being the boron nitride impalpable powder of crystalline random layer structure.
[Claim 3] Engine lubricant according to claim 1 or 2 whose mean diameter of the primary particle of the boron nitride impalpable powder currently distributed in an oil is 0.5 micrometers or less.
[Claim 4] Engine lubricant according to claim 1 to 3 whose amount of the boron nitride impalpable powder currently distributed in an oil is 0.02 - 50 % of the weight.
[Claim 5] Engine lubricant according to claim 1 to 4 which is that in which 50% of the weight or more of the primary particle of said boron nitride impalpable powder observed with an electron microscope has the shape of an abbreviation globular form, and an approximate circle plate configuration.
[Claim 6] Engine lubricant according to claim 1 to 5 whose oil is the oil of a petroleum system, the synthetic oil of ester, or a mixed oil of the oil of a petroleum system, and the synthetic oil of ester.
[Claim 7] Engine lubricant according to claim 1 to 6 with which one or more sorts chosen from a nonionic surface active agent, an anionic surfactant, a cationic surfactant, an amphoteric surface active agent, and an oil solubility surfactant as a dispersant of boron nitride impalpable powder into an oil are added.
[Claim 8] Engine lubricant according to claim 1 to 7 with which one or more sorts chosen from an antioxidant, a viscosity index improver, a pour point depressant, a putrefaction inhibitor, a rusr-proofer, an extreme pressure additive, and a defoaming agent into an oil are added.
[Claim 9] The lubrication approach of the engine characterized by using engine lubricant according to claim 1 to 8.
[Claim 10] Lubricant of the engine characterized by making high concentration distribute in an oil the crystalline random layer structure boron nitride impalpable powder for considering as the engine lubricant which is mixed to commercial engine lubricant and contains the boron nitride impalpable powder of the crystalline random layer structure of an effective dose.
[Claim 11] The lubrication approach of the engine characterized by distributing the crystalline random layer structure boron nitride impalpable powder of an effective dose in an oil, and performing lubrication.

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Drawing selection Representative drawing

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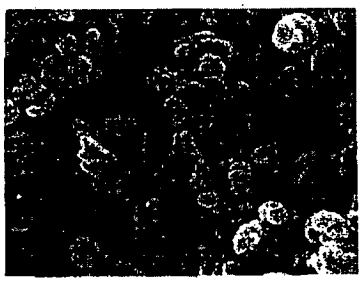

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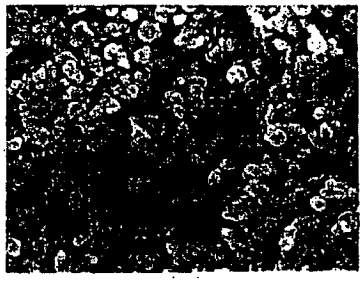

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] This invention relates to the engine lubrication approach at the engine lubricant thru/or the engine lubricant list used for various internal combustion engines' lubrication. This invention relates also to a common lubricating oil, when further exposed to rotation thru/or the slide member of bearing, a gear, and others especially a heavy load thru/or an elevated temperature.
[0002]
[Description of the Prior Art] What added the molybdenum disulfide powder which is a solid lubricant as engine lubricant is proposed. Although the addition effectiveness is accepted in the engine lubricant which added molybdenum disulfide powder, that there is no operating experience so much does not have the remarkable addition effectiveness, and it is presumed that it is for cost to start considering the effectiveness acquired.
[0003] Although boron nitride (BN) is a compound which consists of boron and nitrogen, the polymorphism which has the almost same crystal structure as carbon exists in boron nitride. There are the graphite and cubic system diamonds which have the structure in which the carbon of amorphism and the mesh layer of a hexagon carried out the laminating, such as hexagonal system, in carbon. Graphites, such as hexagonal system which shows cleavability remarkable between the layers which have the structure in which the mesh layer of a hexagon carried out the laminating, show solid-state lubricity among these. The boron nitride of hexagonal system which has the structure in which amorphism boron nitride (henceforth a-BN) and the mesh layer of a hexagon carried out the laminating the bilayer period also in the case of boron nitride (It is hereafter called h-BN), the rhombohedral boron nitride of the structure in which the mesh layer of a hexagon carried out the laminating in a cycle of three layer The random layer structure boron nitride (henceforth t-BN) in which (it being hereafter called r-BN) and the mesh layer of a hexagon carried out the laminating at random, and the cubic boron nitride (henceforth c-BN) of a high-pressure phase are known.
[0004] It is known that there is the same cleavability as the graphite crystal of hexagonal system in a h-BN crystal, and good solid-state lubricity is shown. The lubricative origin of a h-BN crystal is the foundation AWARUSU association same with the case of a graphite crystal with weak association between 2-dimensional hex-steel layers, shows remarkable cleavability in respect of this, and is understood to be because for there to be a property to be easy to slide on the crystal grain child of each other who did cleavage to the shape of a scale between layers.
[0005] It is white and excels in electric insulation, oxidation resistance is higher than a graphite, and since carbon reacts with an iron system ingredient, and it does not melt, or it does not carry out and an iron system ingredient cannot react easily like a graphite, the sintered compact of h-BN powder with high purity has colorlessness or the desirable property in which it is not burned in an iron system ingredient. In this semantics, especially h-BN is the lubricant suitable for the lubrication of a steel system ingredient as solid-state lubricant.
[0006] As an example of the application using the lubricity of h-BN, the lubricating oil which distributed with the homogenizer etc. and was added is proposed [% of the weight / 0.01 - 30] by the fats and oils of ester in boron nitride impalpable powder at JP,61-261397,A. Moreover, the thermal resistance which each mean particle diameter made distribute h-BN powder and polyether ether ketone powder 20 micrometers or less in fluid fats and oils, and the lubricating oil which was excellent in the friction reduction effectiveness are indicated by JP,63-135496,A.
[0007]
[Problem(s) to be Solved by the Invention] Since it is hygroscopic, and it is unstable to the powder of a-BN and is not suitable for it as boron nitride powder added to a lubricating oil, the h-BN powder which does not have hygroscopicity chiefly is used. However, since the price is not cheap about h-BN powder, either, it is used only as a lubricating oil of a special application which is realized although cost increases considerably. Moreover, since the approach which does not pass to have arrived at the

laboratory prototype phase still at last but which can be cheaply compounded with sufficient yield about r-BN or t-BN is not learned, it is the phase before discussing a concrete application.
[0008] The purpose of this invention is to offer engine lubricant thru/or the lubricant for the said addition excellent in the cost performance. Moreover, this invention aims at offering the lubrication approach of the engine using this engine lubricant (or lubricant for the said addition) as another view.
[0009]
[Means for Solving the Problem] In the 1st view of this invention, the engine lubricant of this invention is characterized by carrying out effective dose distribution and containing crystalline t-BN impalpable powder in an oil. In the 2nd view, in an oil, the mean particle diameter of crystalline t-BN impalpable powder 1 micrometer or less and a primary particle distributes h-BN impalpable powder 1 micrometer or less, and the mean particle diameter of a primary particle contains the engine lubricant of this invention, and it is characterized by 50% of the weight or more of boron nitride impalpable powder being crystalline t-BN impalpable powder. Furthermore in the 3rd view, the lubrication approach of the engine of this invention is characterized by carrying out the lubrication of the engine using the engine lubricant containing the crystalline t-BN impalpable powder of an effective dose.
[0010] the diffraction line of the [102] locations which the crystal structure which consists of a 2-dimensional mesh layer in this invention progresses, and show a sharp diffraction line to [004] locations, and mean existence of the regular laminated structure of a mesh layer peculiar to h-BN -- completely -- most **** -- the boron nitride which is not private seal ** is called crystalline t-BN.
[0011] on the other hand, the Mining and Materials Processing Institute of Japan -- it is being explained to Vol.105, No2.p201, and 1989 grades that a-BN is t-BN. However, the powder X diffraction Fig. by the CuK alpha rays of the boron nitride powder currently called t-BN in this reference shows only two broadcloth diffraction lines to the location of [002] diffraction lines of h-BN, ****** [100], and the location of [101] (the characteristic of the diffraction line of h-BN expresses the location of the diffraction line of the powder X diffraction Fig. of the expedient upper boron nitride powder of explanation on these specifications.). there is completely a diffraction line in the same location of [004] diffraction lines below -- it is -- it hardly accepts. The powder X diffraction Fig. of above-mentioned a-BN is the same as the powder X diffraction Fig. of a-BN shown in drawing 1 , and it is not appropriate to suppose that it is above-mentioned a-BN t-BN.
[0012] Boron nitride is chemically stable compared with other solid lubricants, such as a graphite, and there is the description of burning and being hard to reach in ****** and a ferrous material, to about 1000 degrees C in air. The example of the powder X diffraction Fig. of a-BN powder, h-BN powder, and crystalline t-BN impalpable powder is shown in drawing 1 , drawing 2 , and drawing 3 , respectively. If boron nitride is compounded at low temperature 900 degrees C or less, as shown in drawing 1 , the a-BN powder which shows two diffraction lines with wide (broadcloth) width of face to the location of [002] of h-BN of a powder X diffraction Fig. and the location corresponding to the adjoining location of [100] and [101] will be obtained. If this a-BN powder is heat-treated at temperature higher than 1050 degrees C, crystallization will start. If crystallization progresses, the diffraction line corresponding to [002] diffraction lines of h-BN will have small half peak width, and will change to the diffraction line of a strong peak. At this time, [004] diffraction lines also appear as a diffraction line with it in coincidence.
[small half peak width and] [sharp]
[0013]
[Embodiment of the Invention] In addition, in this application, the publication of the numerical range shall represent not only a minimum but the middle any value a top. The any value of 1/10 unit of the predetermined range shall be included especially at least. Crystalline t-BN impalpable powder is specifically heated in the reaction container which held the mixed raw material which contains boric acid alkali, such as sodium borate, as anhydrous boric acid, a urea, and an arbitration component to the non-oxidizing atmosphere. Make it react below 1100 degrees C (preferably 950 degrees C or less), and a-BN is made to generate. Subsequently, it heats at 1200-1500 degrees C (preferably 1200-1400 degrees C, more preferably 1250-1350 degrees C), and can compound by high yield by crystallizing crystalline t-BN (in the condition that boric acid alkali lives together if needed). If the obtained reactant is rinsed

and (being hot water preferably) (acid washing is also included if needed) refined and fusibility components, such as alkali and boron oxide, are removed, the mean particle diameter of a primary particle can manufacture crystalline t-BN impalpable powder 1 micrometer or less by high yield, and can mass-produce it cheaply. In addition, 1450 degrees C or more (if temperature is raised to especially 1500 degrees C or more, h-BN-ization will start, and if it is further made an elevated temperature, h-BN will generate by the quantitative ratio of arbitration.) According to this synthetic approach, by changing the temperature and time amount to crystallize, the particle size of a primary particle can be changed and the boron nitride impalpable powder with which h-BN and crystalline t-BN live together at a various rate can be compounded. This new synthetic approach is explanation ending at Japanese Patent Application No. No. 21052 [nine to] for which it applied previously, and if needed, that detail has a citation in this application, and is used for it.

[0014] It is compounded by the above, and although particle size usually serves as an aggregated particle which the detailed primary particle 1 micrometer or less condensed, if it is made to distribute compulsorily, most can make refined crystalline t-BN impalpable powder the dispersing element of the crystalline t-BN impalpable powder which is a primary particle. mean particle diameter can crack and dissociate distribution if needed even to a detailed primary particle 1 micrometer or less (mean particle diameter is 0.5 micrometers or less, 0.3 micrometers or less, and 0.1 more micrometers or less preferably) by dry grinding, such as wet grinding (quality of a zirconia etc.) using the attrition mill which makes the bead and ball of the ceramics grinding media, a ball mill, other (2 -- formal or 3 types are included) roll-type shear nature mills, etc., or a jet mill. There is no hygroscopicity which is looked at by a-BN powder in this crystalline t-BN impalpable powder, it is stable and there is also oxidation resistance. According to the above-mentioned manufacture approach, the crystalline boron nitride impalpable powder which offer of the impalpable powder which has the same particle size distribution is possible also about h-BN, and contains h-BN partially and which mainly consists of crystalline t-BN can also be mass-produced. Crystallization to h-BN is industrially realizable by carrying out predetermined time heat treatment of crystalline t-BN above 1500 more degrees C (further 1600-1800 degrees C, 1750-1800 etc. degrees C to [Preferably] about 1850 degrees C, etc.). Each primary particle of the h-BN impalpable powder obtained by this manufacture approach and crystalline t-BN impalpable powder consists of a detailed crystal which has cleavability, and h-BN impalpable powder and crystalline t-BN impalpable powder, especially crystalline t-BN impalpable powder show the further excellent solid-state lubricity.

[0015] The reason the mean particle diameter of a primary particle uses as impalpable powder 1 micrometer or less boron nitride impalpable powder which engine lubricant is made to distribute in the specific view of this invention is for being easy to enter narrow space a certain forge fire, if boron nitride impalpable powder is detailed, and being easy to demonstrate the function as engine lubricant. The mean particle diameter of the primary particle of the boron nitride impalpable powder distributed in an oil excels [one / finer] in lubricity, and its thing (0.5 micrometers or less, 0.3 micrometers or less or 0.2 micrometers or less, and 0.1 more micrometers or less) is especially desirable. Since the cohesive force dissociates to a primary particle or a smaller aggregated particle gradually so greatly according to the shearing force at the time of the use as engine lubricant and the detailed primary particle of especially crystalline t-BN is distributed even if it carries out the configuration of the aggregated particle, if what has a small particle size of a primary particle is used, the function as good engine lubricant can be demonstrated. As a standard of the particle size of an aggregated particle, it is thought further that what is necessary is to consider that the mesh of an engine-oil strainer does not carry out blinding if needed, and just to set that what is necessary is just 10 micrometers or less usually. [0016] Moreover, as for the boron nitride impalpable powder distributed in engine lubricant, it is desirable that it is the boron nitride impalpable powder which contains crystalline t-BN impalpable powder 50% of the weight or more. With the crystalline t-BN impalpable powder said to this invention, typically Crystallization of a 2-dimensional mesh layer While each half peak width of the diffraction line corresponding to the diffraction line which is progressing and is in the [002] locations of a h-BN crystal and [004] locations serves as a small (the half peak width of [004] diffraction lines which have 2theta of a powder X

diffraction Fig. obtained by CuK alpha rays in abbreviation ** is 0.6 degrees or less) sharp diffraction line [102] diffraction lines peculiar to the h-BN crystal in which it is shown that regularity is in a laminated structure mean especially the thing from which most or the diffraction line corresponding to [do not accept at all and] [100] diffraction lines and "101" diffraction lines of h-BN is one diffraction line ([10] diffraction lines of t-BN). That it is the X diffraction line of the pattern which a side dwindles whenever [angle-of-elevation / of [10] diffraction lines of this t-BN] means that it is crystalline t-BN of the random layer structure which does not have regularity in how (pattern of a laminating) to pile up a hex-steel layer, although 2-dimensional crystallization is progressing. That it is crystalline t-BN in this invention [100] of the powder X diffraction Fig. of a h-BN crystal typically, It can specify numerically as a thing with the relation of S102/(S100+S101) <=0.02 among the area (it is proportional to the reinforcement of each diffraction line) S100, S101, and S102 which each diffraction line corresponding to the diffraction line of [101] and [102] occupies.

[0017] As for the boron nitride impalpable powder distributed in the oil of engine lubricant, it is desirable that the mean particle diameter of the aggregated particle which carried out distributed processing with wet using media, such as alcohol with sufficient dispersibility, uses impalpable powder 5 micrometers or less (3 more micrometers or less, 2 micrometers or less, more preferably 1 micrometer or less). A crack progresses gradually while in use, it becomes a primary particle further, the rate of a primary particle increases gradually, and the aggregated particle of the boron nitride impalpable powder in engine lubricant comes to demonstrate a detailed aggregated particle and the more excellent lubricity. therefore, extent which exists in early stages -- you may be engine lubricant with which the boron nitride impalpable powder containing many large aggregated particles was distributed. As crystalline t-BN impalpable powder used for the engine lubricant of this invention, the mean particle diameter of a primary particle is very as fine as 0.5 micrometers or less, 0.2 micrometers or less, and 0.1 more micrometers or less (nano meter order), and what has uniform particle size distribution (particle size gathered) is suitable.

[0018] With engine lubricant, although crystalline t-BN impalpable powder shows lubricity also with the dry impalpable powder, it mixes with an oil and it is distributed in an oil so that boron nitride impalpable powder can be sent into the narrow part by which lubrication is made. However, BN impalpable powder is stored with fine particles, and the need is accepted, and into an oil, it can distribute and mix and can also use. As an oil of engine lubricant, the oil of a petroleum system, the synthetic oil of ester with good dispersibility, or the mixed oil of the oil of a petroleum system and the synthetic oil of ester can be used, and an oil should just select the optimal combination according to the purpose and conditions of use.

[0019] In addition, as a practical use preservation gestalt, it can be dealt with in the condition of having distributed to high concentration, as lubricant for addition (or master liquid), and actual engine lubrication can be performed by adding master liquid to a specified quantity usual engine oil. However, distributed content of the predetermined BN impalpable powder can be carried out into an engine oil from the beginning. There is especially no limit in the concentration of BN that what is necessary is just to define the distributed condition as lubricant for addition in consideration of that that dispersible nature is securable, sedimentation, and condensation do not arise, a fluidity being securable, etc. at the time of the addition to an engine oil. Although 0.1 - 50 % of the weight, 1 - 30 etc. % of the weight of the amount of BN(s) in the lubricant for addition, etc. are that standard preferably to 70 % of the weight about above an effective dose, by the engine performance of the grain size of the liquids and solutions to be used, a dispersant, etc., and the quantitative ratio, this range is adjustable suitably and the concentration of BN can be selected according to the purpose of use.

[0020] Since crystalline t-BN thru/or h-BN are stable, it can use the engine lubricant marketed for the oil of the engine lubricant of this invention as it is. It is desirable to add as a dispersant one or more sorts chosen from a nonionic surface active agent, an anionic surfactant, a cationic surfactant, an amphoteric surface active agent, and an oil solubility surfactant to engine lubricant so that boron nitride impalpable powder can be maintained at a stable suspension condition. To commercial engine lubricant, control the generation and ****** of products of combustion which are generated in a lubricating oil at the time of

use, or The dispersant added for the purpose of raising lubricating properties, detergent, an antioxidant, Various additives, such as corrosion inhibitor, a rust preventives, an oily agent, an extreme pressure additive, an oil film reinforcement, an antiwear agent, a pour point depressant, a viscosity index improver, and a defoaming agent, are contained. Also about the engine lubricant of this invention It is desirable to add one or more sorts chosen from an antioxidant, a viscosity index improver, a putrefaction inhibitor, a rusr-proofer, an extreme pressure additive, and a defoaming agent.

[0021] It is desirable to add an above-mentioned dispersant or an above-mentioned surface active agent, to scour with a roll or to carry out preferential grinding to the bottom of an operation of shearing force in a ball mill or an attrition mill, in order to make homogeneity distribute boron nitride impalpable powder and especially detailed crystalline t-BN impalpable powder in an oil. [carrying out high-speed churning with a homogenizer] Since it will not be bulky on the occasion of conveyance or preservation if engine lubricant is beforehand prepared in the condition of the suspension of desirable deep boron nitride impalpable powder, is saved in it, and it is used from it, thinning with an oil so that it may become the concentration needed on the occasion of use, it is convenient. In the engine lubricant of this invention, boron nitride impalpable powder demonstrates the lubricity excellent also in such little addition that it is detailed crystal powder.

[0022] Although the reason the engine lubricant which made the crystalline t-BN impalpable powder with which the 2-dimensional crystal structure progressed suspend shows good lubricating properties compared with the lubricating oil which made h-BN impalpable powder suspend is not clear, it is thought that the difference (fundamentally random in crystalline t-BN) of the regularity of the laminating of a mesh layer is the main factor at least. That is, by crystalline t-BN, since the bond strength between hex-steel layers is smaller than the bond strength between the hex-steel layers of a h-BN crystal, it is easy to slide between layers, or it is easy to carry out cleavage between layers and there is no directivity in the direction parallel to the hex-steel layer of a crystal, it is thought that the crystal of the shape of a scale of cleavability is easy to slide in any direction parallel to a mesh layer. Furthermore, it is thought that it is because the particle of the boron nitride the primary particle is detailed (obtained as impalpable powder of an aggregated particle with a particle size of 10 micrometers or less which consists of a detailed primary particle of the nano meter order which is usually the mean particle diameter of 1 micrometer or less, 0.3 micrometers or less, and 0.1 more micrometers or less), detailed, and crystallinity tends to demonstrate the function as a solid lubricant in the crystalline t-BN impalpable powder compounded by the above-mentioned manufacture approach. [boron nitride]

[0023] The boron nitride impalpable powder which has temperature in case crystalline boron nitride impalpable powder crystallizes preferably 1100 degree C or less of a-BN compounded at low temperature 950 degrees C or less, and extent of various crystallization by time amount is obtained. 1200-1500 degrees C of 1200-1400 degrees C of crystallization to t-BN are more preferably advanced at 1300**50 degrees C. If temperature is raised further and it is made to crystallize, finally boron nitride will be converted into stable h-BN at an elevated temperature. If it heat-treats above 1450 degrees C, the inversion to h-BN will start, and crystalline t-BN impalpable powder turns into powder with which t-BN and h-BN were intermingled. The boron nitride impalpable powder distributed in engine lubricant demonstrates the lubricity excellent in the direction with many content rates of crystalline t-BN impalpable powder. 50% of the weight or more of the boron nitride impalpable powder preferably contained in a lubricating oil in order to demonstrate the outstanding lubricity, more than (70 % of the weight, 80 % of the weight or more, 90 % of the weight or more, 95 more % of the weight or more, and 99 % of the weight or more -- substantial -- all --) -- it is desirable to consider as crystalline t-BN impalpable powder. The content rate of the crystalline t-BN impalpable powder in boron nitride impalpable powder can measure the reinforcement (area which a diffraction line has) of the diffraction line obtained according to a powder X diffraction, when a mixed rate measures the reinforcement of the powder X diffraction of the boron nitride mixing powder Fig. of a known criterion.

[0024] h-BN and crystalline t-BN impalpable powder show the good lubrication effectiveness also with an addition with little finer one. For this reason, as for the mean particle diameter of the aggregated particle of the boron nitride impalpable powder in a lubricating oil, it is desirable to be referred to as 10

micrometers or less, 5 micrometers or less, and 2 more micrometers or less (especially preferably 1 micrometer or less). If boron nitride impalpable powder is ground in mills, such as an attrition mill, an aggregated particle can be cracked comparatively easily even to the impalpable powder which mainly consists of a primary particle. Being able to measure the particle size distribution of boron nitride impalpable powder with a sedimentation method, in this invention, mean particle diameter says the particle size of the location whose addition weight of weight addition particle size distribution is 50 % of the weight. Moreover, in order to ask for the mean particle diameter of a primary particle, the enlargement of the scanning electron microscope (SEM) of impalpable powder is taken and measured.

[0025] If the mean particle diameter (secondary particle size) of boron nitride impalpable powder is as fine as 2 micrometers or less and 1 more micrometer or less, many of particles of boron nitride impalpable powder will be made detailed by even the primary particle, and the lubricity of a lubricating oil will improve further by this. Since fine boron nitride impalpable powder can enter the small hollow of the micron order of a sliding surface, when the mean particle diameter of an aggregated particle distributes 1 micrometer or less, 0.5 more micrometers or less, and fine impalpable powder, it can give still better lubricity into engine lubricant.

[0026] Since the amount of the boron nitride impalpable powder distributed in engine lubricant can cover an extensive service condition while being able to give the good lubrication effectiveness, although a suitable and economical content exists according to the service condition, as for the variance of the boron nitride impalpable powder in engine lubricant, it is desirable to consider as 0.02 - 50 % of the weight. What will be made uniform suspension if the lubrication effectiveness that the amount of mixing is obtained at 0.02 or less % of the weight does not clarify but it mixes exceeding 50 % of the weight is disagreeable ****** which becomes difficult. In order to demonstrate lubricity with a sufficient cost performance, if it is desirable to make the amount of mixing of boron nitride impalpable powder into 0.05 - 20 % of the weight and further 0.1 - 10 % of the weight and it responds to an application, 5 or less % of the weight or 2 % of the weight or less, and 1 % of the weight or less are sufficient.

[0027] The configuration of a primary particle presents the shape of approximate circle tabular or an abbreviation globular form, and the crystalline t-BN impalpable powder with which the 2-dimensional crystal structure progressed has the outstanding lubrication engine performance so that the SEM enlargement of drawing 5 may see. the primary particle of the boron nitride impalpable powder preferably contained in a lubricating oil with the lubricating oil of this invention since addition of crystalline t-BN impalpable powder can give lubricating properties excellent in engine lubricant -- 70 % of the weight or more (80 more % of the weight or more, 90 % of the weight or more, most preferably substantially wholly) has the shape of approximate circle tabular or an abbreviation globular form still more preferably 50% of the weight or more. The particle size of the primary particle of the crystalline t-BN impalpable powder which can observe the configuration of the primary particle of boron nitride impalpable powder with the enlargement of SEM, and is obtained by the above-mentioned manufacture approach is usually as small as 1 micrometer or less. It is understood that the primary particle of crystalline t-BN impalpable powder does not become hexagon-head tabular like the crystal grain child of h-BN because crystalline t-BN does not have regularity in the laminating relation between the layers of a 2-dimensional mesh layer.

[0028] The oil of engine lubricant can choose a suitable thing according to a service condition. It is [that what is necessary is just the oil which has the good dispersibility over boron nitride impalpable powder, or the oil which can give good dispersibility by adding a dispersant] desirable to use the oil of a petroleum system cheap as an oil, the synthetic oil of ester with sufficient dispersibility, or the mixed oil of the oil of a petroleum system and the synthetic oil of ester. It is desirable to use commercial engine lubricant or an oil equivalent to commercial engine lubricant from the ability of a commercial item to obtain cheaply as the example.

[0029] It cannot be said that the boron nitride impalpable powder itself not necessarily has the good dispersibility over an oil. Moreover, even if boron nitride impalpable powder is fine, in the condensed impalpable powder, it is stabilized and good lubricity cannot be demonstrated. In order to distribute

boron nitride impalpable powder in an oil and to demonstrate lubricity, it is desirable to add a dispersant in engine lubricant. As a result of carrying out comparison examination of the various dispersants, it admitted that especially a nonionic surface active agent, an anionic surfactant, an amphoteric surface active agent, and an oil solubility surfactant were useful for distributing boron nitride impalpable powder in an oil. That is, it is desirable to use one or more sorts chosen from a nonionic surface active agent, an anionic surfactant, an amphoteric surface active agent, and an oil solubility surfactant as a dispersant of the engine lubricant of this invention. If the dispersant is added to engine lubricant, re-distribution is easy even if boron nitride impalpable powder may sediment in an oil according to a specific gravity difference with an oil. Especially desirable dispersants are an amphoteric surface active agent and an oil solubility surfactant among these dispersants.

[0030] It is desirable to mix various kinds of additives other than a dispersant according to the conditions used for the engine lubricant of this invention and the class of oil. As a concrete example of an additive, there are an antioxidant, a viscosity index improver, a pour point depressant, a putrefaction inhibitor, a rusr-proofer, an extreme pressure additive, and a defoaming agent. The well-known additive added as these additives by the engine lubricant marketed can be used preferably.

[0031]

[Example] Hereafter, although an example explains the engine lubricant of this invention concretely, the following examples are examples of this invention and the engine lubricant of this invention is not limited to the example explained below. That is, the engine lubricant of this invention can be combined with the various conditions of the class displacement of the engine which carries out lubrication, a rotational frequency, torque, and temperature and others, and can be applied according to the various views.

[0032] [Example 1] (manufacture of crystalline t-BN impalpable powder)

3.5kg (B-2 O3) of [example 1A] anhydrous boric acid, 5.3kg (NH2) (2CO) of ureas, Mixture which consists of 0.63kg (Na2B4O7.10H2O) of borax is used as a start ingredient. This mixture is put into the reaction container made from stainless steel with a diameter of 530mm with a lid. This reaction container is put in in a furnace, it applies to a multistage story (250-500 degrees C; 500-600 degrees C, 600-700 degrees C, 700-800 degrees C, and 800-900 degrees C) 10 minute each, and a temperature up is carried out to it, and it held for 10 minutes and was made to react at 900**10 degrees C (a total of 1 hour). The steam blew off at about 100 degrees C, a component began to fuse partially at 200 degrees C at first, the reaction progressed, and, even in the bubble, emission of gas continued foamily. The steam was mainly emitted to further 350-400 degrees C, and when held for 10 minutes at 900 degrees C, emission of generation gas decreased. It cooled radiationally in this condition, the lid of a reaction container was opened, and the reactant was taken out from the reaction container. At this time, the reactant in a reaction container had become the shape of a Japanese brittle of dry BASABASA which shows that B-2 O3 carried out the completion of a reaction mostly. The reactant was cracked in the reaction container and it took out by vacuum suction, and it applied to the grinder (crusher) further, and ground, and the powder of 1mm pass was obtained (above, primary process). Let this generation powder below be the start ingredient of a secondary process.

[0033] It moved to the container with a lid (a lid is a closedown lightly) made from ceramic (alumina) refractories, and inserted in the electric furnace the whole container with a lid. N2 or CO2 was introduced into the electric furnace, and it considered as the non-oxidizing atmosphere, it raised from ordinary temperature, having applied it to 1300 degrees C for 10 hours, and finally, it held at about 1300 degrees C for 2 hours, and cooled radiationally at them.

[0034] It washed having harmonized among 80-85-degree C ion, and fully carrying out stirring grinding of the powder picked out from the container with a lid with exchange water (hot water), and except for the alkali component, finally it washed from the acid (HCl), neutralized, rinsed further, and dried after that. About 0.6-0.65kg t-BN was obtained per 10kg of start raw materials of a secondary process, and after washing. This became about 28.5% or more of t-BN to the start boron weight of a primary process, and yield was 70% or more of high rate, and, moreover, was a high grade. In addition, 10 - 20% of weight loss was accepted from a primary process product to heat treatment of a secondary process.

[0035] Although it was a sample different from [example 1B] example 1A, the crystalline t-BN powder obtained almost like example 1A was investigated by the powder X diffraction by CuK alpha rays. The X diffraction Fig. of the obtained synthetic powder is shown in drawing 3 . If the powder X diffraction Fig. of drawing 3 is compared with the well-known X-ray diffraction pattern of h-BN shown in drawing 2 , a diffraction line with the boron nitride of the powder X diffraction Fig. of drawing 3 sharp in the location corresponding to [considerable t-BN crystallization is progressing and] the diffraction line and [004] diffraction lines of [002] of drawing 1 will be accepted in about 26.6 degrees and about 55 degrees, respectively. [of h-BN] However, it turns out that a diffraction line is not accepted in the location corresponding to [102] diffraction lines of h-BN. Moreover, a quite sharp diffraction line is in the location (41.55 degrees) corresponding to [100] diffraction lines of h-BN. This [100] diffraction line is a side whenever [with [101] sharp diffraction lines of h-BN / angle-of-elevation], and has lapped at [101] low diffraction lines and the low skirt, and [101] diffraction lines are drawing the background which was a side whenever [angle-of-elevation], lengthened the skirt and increased a little. This [101] diffraction line does not exist as a sharp projection. This means that this synthetic boron nitride powder is t-BN powder with high purity with which crystallization progressed. The powder of drawing 3 is an example of the crystalline t-BN powder said to this invention.

[0036] When the location and half peak width of a powder X diffraction Fig. of 2theta were investigated, [002] diffraction lines were in 26.58 degrees, [004] diffraction lines were in 55.0 degrees, and half peak width was 0.47 degrees. [of drawing 3] [of each diffraction line] This shows that half peak width of a h-BN[004] considerable diffraction line is made to about 0.5 degrees or less.

[0037] The enlargement (x20000 time and x10,000 time) by the sample SEM of the t-BN impalpable powder obtained like [example 1C] example 1A is shown in drawing 5 . The mean particle diameter of the primary particle of this t-BN composition powder is about 0.45 micrometers, and the SEM photograph of drawing 5 R> 5 shows that the particle size of a primary particle exists within the limits of 0.3-0.75 micrometers substantially. moreover, disc-like [which this primary particle does not show a hexagon-head tabular crystal grain child configuration peculiar to the hexagonal system looked at by the primary particle of h-BN, but is considered to be peculiar to a crystalline t-BN crystal] (big thing) thru/or abbreviation -- it admitted being spherical (small thing).

[0038] t-BN powder was compounded like example 1A except having added 1% of the weight in the raw material by sotogake by using as seed crystal the t-BN powder which compounds by the same approach as [example 1D] example 1A, and contains many dispersed primary particles. In this example, advance of first order reaction also became early and much more improvement was accepted in the yield of last generation t-BN. In addition, the yield of the generation BN to preparation anhydrous boric acid reaches to a maximum of 80% or more.

[0039] The dispersing element of the t-BN powder created on the same conditions as [example 1E] example 1A is created, particle-size-distribution measurement is performed, and the result is shown in drawing 4 . Measurement is HORIBA. It carried out using the LA-700 particle-size analyzer. As a result, the median size of 0.30 micrometers, 95.2% of accumulation not more than particle diameter 1micrometer, and 90% particle diameter were 0.75 micrometers. In addition, if the point (measured condensing considerably) which cannot be said to be a perfect primary particle in this measurement is ****(ed), it is trustworthy that it is an average of 0.3 micrometers or less. In addition, the specific surface area was 23.4m2/cm3. In addition, the SEM photograph of another sample obtained similarly is shown in drawing 6 . The particle is carrying out the shape of approximate circle tabular thru/or ****, and the first [an average of] particle diameter is about 0.3 micrometers, and it turns out that the particle size of a primary particle is within the limits of [very narrow] 0.2-0.45 micrometers substantially.

[0040] Without having changed the mixing ratio of [example 1F] anhydrous boric acid and a urea into 4:9 (weight ratio), and using borax, the gas drainage hole of a well-closed container was fully made into the example 1A said appearance except having changed the interior into the pressurization condition, as for the crimp by making primary process heating into 1.5 hours, and holding terminal temperature for 15 minutes at 920-950 degrees C, and BN was compounded. The secondary process was performed on the almost same conditions as example 1A, and performed washing similarly. t-BN of a high grade was

obtained extremely. The SEM photograph is shown in drawing 7 . A configuration is an abbreviation globular form, and the first [an average of] particle diameter is about 0.25 micrometers, and it turns out that the diameter of a primary particle has most in the range of 0.15-0.38 micrometers (namely, about 0.1-0.4 micrometers) substantially by 0.2-0.3 micrometers. In addition, although 4:6-4:9 were desirable as for the mixing ratio (weight ratio) of anhydrous boric acid and a urea, 4:9 gave the best result.
[0041] The X diffraction Fig. of sample t-BN created on the same conditions as [example 1G] example 1F is shown in drawing 8 . If the powder X diffraction Fig. of drawing 8 and drawing 2 is compared, a diffraction line with the boron nitride of the powder X diffraction Fig. of drawing 8 sharp in the location corresponding to [considerable t-BN crystallization is progressing and] the diffraction line and [100] diffraction lines of [002] of drawing 2 will be accepted in 26.7 degrees and 41.8 degrees, respectively. [of h-BN] However, it turns out that the location of the diffraction line of [002] is shifted to the side whenever [angle-of-elevation] a little compared with the correspondence diffraction line position of h-BN, and a diffraction line is not accepted in the location (50 degrees) corresponding to [102] diffraction lines of h-BN at all. Moreover, although it is not so high in the location (41.8 degrees) corresponding to [100] diffraction lines of h-BN, a sharp diffraction line is in it. this diffraction line should pass a shoulder in a side whenever [with [101] diffraction lines of h-BN / angle-of-elevation] -- a little long skirt is lengthened -- **** (henceforth (10) diffraction lines) -- [101] diffraction lines do not exist as a clear projection. This means that this synthetic boron nitride powder is single phase t-BN powder with high purity with which crystallization as t-BN progressed. The powder of drawing 8 is an example of the high grade crystallinity t-BN powder said to this invention (especially super-submicron thing of 0.2-0.3-micrometer order). It is enough imagined from the lowness of a background that it is a high grade and that it is t-BN single phase. That is, the point that the peak drawing 3 and the diffraction line of drawing 8 indicate B-2 O3 to be has not appeared at all will attract attention.
[0042] [Example 2] (preparation and the practical use test of engine lubricant)
It is the following, and [example 2A] engine lubricant was made and prepared. Namely, lubricating oil base oil (the mixed liquor which consists of a petroleum system, about 218 degrees C (coc) of flash points, the kinematic viscosity abbreviation 27.8mm2/s(cSt) 64 weight section in 40 degrees C, and the polyoxyethylene coconut alkylamine derivative (Kao AMITO 102) 6 weight section was put into the pot mill made from an alumina with a capacity of 7l. with with a diameter of 10mm alumina-balls 7.6kg, and it mixed by 60rpm for 1 hour.) Next, the crystalline t-BN impalpable powder 30 weight section was added in the same pot mill container made from an alumina, and the suspension (master liquid) containing crystalline t-BN impalpable powder with a particle size [first / an average of] of 0.3 micrometers which used and manufactured about 30% of the weight of borax in 1l. which mixed and ground by 60rpm for 24 hours, and homogeneity was made to distribute was obtained. It considered as the engine lubricant which dilutes with engine lubricant A (bar screw neo SJ15 made from PETERORUBU International W-40) of marketing of this suspension about 30 times, and contains t-BN impalpable powder 1.0% of the weight. In addition, in commercial engine lubricant A, a small amount of antioxidant as an additive, the viscosity index improver, and the rusr-proofer are added.
[0043] The obtained engine lubricant containing crystalline t-BN impalpable powder was put into the engine of Sedan made from the Fuji **** (vehicle name legacy, 2,000 cc displacement EFI-MT vehicle), the oil filter was exchanged for the new article at coincidence, and the driving test was carried out. As a result of performing a driving test by the commutation root of about 37km of one way including 15km highway transit, at the time of high-speed transit, it was especially admitted at the time of an idling and transit that an engine rotational noise decreased clearly. Moreover, since acceleration nature improved, it could judge that engine horsepower improved, and the average mileage per 1l. of regular gasoline was extended from 6.8km (at the time of use of commercial engine lubricant A) to 8.9km. Furthermore, as a result of continuing and using the engine lubricant same after that succeedingly for the lubrication of the engine of this Sedan, the durability of engine lubricant itself is also extended and it was judged that it could be equal also to transit exceeding 20,000km. Moreover, although engine lubricant was sampled and the oil filter was checked, abnormalities were not accepted at all.

[0044] [Example 2B] (preparation and the practical use test of engine lubricant)
Using crystalline t-BN (primary particle diameter of about 0.25 micrometers) manufactured without using borax, this crystalline t-BN impalpable powder 30 weight section was mixed with the lubricating oil base oil 64 weight section and the polyoxyethylene coconut alkylamine derivative 6 weight section like example 2A, and the suspension (master liquid) which contains about 30% of the weight of crystalline t-BN impalpable powder in 1l. was obtained. The engine lubricant which is mixed to engine lubricant B (the Nippon Oil Co., Ltd. make, trade names ZOA and SG, 10W-30) of marketing of this suspension, and contains t-BN impalpable powder 1% of the weight was obtained. This engine lubricant was put into the engine of the RV (vehicle name PAJIERO, 3,500 cc displacement ECI-automatic-transmission car) by MITSUBISHI MOTORS CORP., the oil filter was exchanged for the new article at coincidence, and the driving test was carried out. As a result of performing a driving test in the transit (about 45% is highway) section which uses a highway abundantly, it was admitted at the time of an idling and transit that the rotational noise of the engine especially at the time of high-speed transit decreased clearly. Moreover, when engine horsepower improved, acceleration nature improved, and to the mileage per l. by the regular gasoline at the time of commercial engine lubricant B use having been 6.2km, when this engine lubricant containing crystalline t-BN impalpable powder was used, mileage was extended to 7.8km.
[0045] [Example 2C] (preparation and the practical use test of engine lubricant)
The mixed liquor which consists of the lubricating oil base oil (petroleum system, about 218 degrees-C [of ignition], kinematic viscosity about 27.8mm2/s in 40 degrees C (cSt)) 64 weight section and the polyoxyethylene coconut alkylamine derivative (Kao AMITO 102) 6 weight section was put into the container of the pot mill made from an alumina with a capacity of 7l. with with a diameter of 10mm alumina-balls 7.6kg, and it mixed by 60rpm for 1 hour. Next, Example 3 and this procedure compounded and refined and the suspension (master liquid) which 30% of the weight of crystalline t-BN impalpable powder distributed to homogeneity in the oil like example 2A was obtained.
[0046] The engine lubricant which is mixed to the diesel-power-plant lubricating oil C of marketing of this suspension (an antioxidant, a viscosity index improver, and a rusr-proofer are included as the trade name ZOA diesel RV by Nippon Oil Co., Ltd., CF-SAE15W-40, and an additive), and contains crystalline t-BN impalpable powder 1% of the weight was obtained. This engine lubricant containing crystalline t-BN impalpable powder was put into the diesel power plant of the small freight car (the Hilux pickup, 2,800 cc displacement MT vehicle with a turbocharger) by Toyota Motor Corp., the oil filter was exchanged for the new article at coincidence, and the driving test was carried out. As a result of performing a driving test in the transit (about 20% is highway) section which uses a highway abundantly, it was admitted at the time of an idling and transit that the rotational noise of the engine especially at the time of high-speed transit decreased clearly. Moreover, acceleration nature improved, and also to the mileage per 1l. of gas oil when using the commercial diesel-power-plant lubricating oil C having been 9.5km, when the engine lubricant containing crystalline t-BN impalpable powder of this invention was used, mileage was extended to 10.9km.
[0047]
[Effect of the Invention] With the synthetic technique of the conventional boron nitride, the mass-production approach which can be cheaply offered with the sufficient yield also about h-BN powder did not exist as well as crystalline t-BN impalpable powder. For this reason, it was expensive also about h-BN powder, and the application as a lubricating oil was also limited only to the ********** application. However, by having established the above-mentioned synthetic technique, as well as especially h-BN powder, the crystalline t-BN impalpable powder excellent in solid-state lubricity is mass-produced cheaply, and can be offered now. This invention was applied to engine lubricant by the expansion to the new application of the t-BN impalpable powder which is not conventionally known ignited by establishment of said synthetic technique, and this invention, and it checked that remarkable effectiveness was acquired by addition of the crystalline t-BN impalpable powder to engine lubricant. That is, when using the engine lubricant of this invention for engine lubrication, while the engine rotational noise decreased clearly, the useful life longevity of elongation and engine lubricant itself

extended [the mileage per 1l. of fuels (fuel consumption)] notably, and it checked that it was satisfactory also about the cooling effect of the engine by engine lubricant. If based on these test results, naturally the endurance of an engine should also be extended. Therefore, the frictional resistance in the interior of an engine can be reduced notably, and especially reduction of an engine rotational noise is further raised notably in the silence of the vehicle interior of a room at the time of high-speed transit, and is made to improve the durability of fuel consumption and an engine notably at the time of starting and acceleration by using the engine lubricant containing crystalline t-BN impalpable powder of this invention for engine lubrication. The utility value on the industry of the engine lubricant of this invention is great in this way. Furthermore, it cannot be overemphasized that the lubricating oil of this invention demonstrates the engine performance which was excellent also as a common lubricating oil to rotation thru/or the slide member of bearing, a gear, and others.

[Translation done.]